

## (12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date  
12 February 2004 (12.02.2004)

PCT

(10) International Publication Number  
WO 2004/013267 A2

(51) International Patent Classification<sup>7</sup>:

C11D

(74) Agents: BROWN, Andrew, Stephen et al.; Reckitt Benckiser plc, Group Patents Department, Dansom Lane, Hull HU8 7DS (GB).

(21) International Application Number:

PCT/GB2003/003425

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(22) International Filing Date: 5 August 2003 (05.08.2003)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:  
0218194.9 6 August 2002 (06.08.2002) GB

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

(71) Applicant (for all designated States except MN, US): RECKITT BENCKISER N.V. [NL/NL]; Kantoorgebouw De Appelaer, De Fruittuin 2-12, NL-2132 NZ Hoofddorp (NL).

(71) Applicant (for MN only): RECKITT BENCKISER (UK) LTD [GB/GB]; 103-105 Bath Road, Slough, Berkshire SL1 3UH (GB).

(72) Inventors; and

(75) Inventors/Applicants (for US only): DE DOMINICIS, Mattia [IT/IT]; Reckitt Benckiser Italia, Piazza S. Nicolo 12/3, I-30034 Mira (IT). RIGHETTO, Zeffirino [IT/IT]; Reckitt Benckiser Italia, Piazza S. Nicolo 12/3, I-30034 Mira (IT).

## Declarations under Rule 4.17:

- as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii)) for all designations
- of inventorship (Rule 4.17(iv)) for US only

## Published:

- without international search report and to be republished upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: SOLID FORMULATIONS

(57) Abstract: The invention relates to a method of cleaning carpet, the method comprising the addition to the reservoir of a carpet cleaning machine a water-soluble solid product comprising a carpet cleaning composition, adding water to the reservoir of the carpet cleaning machine prior to or after adding the solid product, and operating the carpet cleaning machine on a solid carpet.

SOLID FORMULATIONS

The invention relates to a method of cleaning carpet, the method comprising the addition to the reservoir of a carpet cleaning machine a water-soluble solid product

10 comprising a carpet cleaning composition, adding water to the reservoir of the carpet cleaning machine prior to or after adding the solid product, and operating the carpet cleaning machine on a soiled carpet.

15 The present invention relates to water-soluble solid products for carpet cleaning machines, preferably in the form of a powder, granule, pellet or tablet, and methods of making such a solid product and methods of using such a product. Preferred solid products are tablets that have

20 been specifically designed for carpet cleaning and especially for carpet cleaning extraction machines. The solid products of the invention need to be soluble in water and to dissolve in a short time period, typically less than 5 minutes, without any stirring, to produce a

25 product having low or no solid residue. A common feature of many carpet cleaning machines is the inclusion of a fine mesh which acts as a filter preventing solid material from entering and damaging the pumping mechanisms. Therefore, it is important that there are no

30 solid residues in the reservoir, so as to avoid any blockages.

US 20020082187 describes the use of expanded sodium perborate as a carrier for liquids, to be used for

35 cleaning carpets. In passing it is mentioned that the composition may be presented as a tablet.

5 We present as a feature of the invention a method of  
cleaning a soiled carpet the method comprising adding to  
the reservoir of a carpet cleaning machine a water-  
soluble solid product comprising a carpet cleaning  
composition, adding water to the reservoir of the carpet  
10 cleaning machine prior to or after adding the solid  
product, and operating the carpet cleaning machine on the  
soiled carpet.

15 Preferably the solid product is added to the reservoir of  
the carpet cleaning machine prior to adding the water.

The solid product may be a powder, granule, pellet or a  
tablet. Preferably the solid product is a tablet.

20 Preferably a combination of effervescent technology,  
which uses a carbonate/bicarbonate with an acid, and  
optionally a swelling disintegrant is used to obtain  
rapid dissolution. The pre-dosed solid product is put  
into the carpet cleaner machine tank with water. The  
25 product dissolves quickly so that it is possible to start  
cleaning in a short period of time without danger of  
blocking the machine.

30 The product provides the consumer with a convenient solid  
form, which is pre-dosed when in tablet form, avoiding  
the need to dilute the product before it is added to the  
machine, which may be necessary with liquid concentrate  
formulations.

35 A problem associated with cleaning carpets is the high  
repellancy of the carpet to water. This is primarily

5 caused from two sources, the first being the amount of soiling which can accumulate on carpets and, secondly, the prevalence of stain repelling treatments which are increasingly commonly applied to carpets either during manufacture or by the consumer. In this invention a  
10 "super wetting" agent is added to the composition and is a special surfactant added at levels of below 10%w/w of the composition, preferably below 9, 8, 7, 6 or 5% w/w, of the composition. It is preferred that at the levels described above, and in combination with any other  
15 surfactant present, it is able to lower the surface tension of the final liquid cleaning formulation to values below 28 mN/m, when 1 to 25g, ideally 2 to 12g, preferably 3 to 10g, or 15g of the solid composition is dissolved in 4 litres of water.

20 We present as a further feature of the invention a water-soluble carpet cleaning composition in solid form comprising at least one surfactant and at least one super wetting agent wherein the combined effect of the  
25 surfactant and the super wetting agent in the composition is capable of reducing the surface tension of water below 28 mN/m when 1 to 25g, ideally 15g of the composition is dissolved in 4 litres of water.

30 We have developed a product that contains a composition particularly suitable for carpet cleaning extraction machines. These compositions comprise at least one surfactant and at least one super wetting agent. Preferably these compositions additionally comprise from  
35 5 to 50%, 10 to 50%, 12 to 45% w/w of at least one of the following: a builder, an antifoaming agent, a dispersing

5 agent or an antiresoiling agent. The composition may also compare up to 15%, 10%, 8%, 6%, 4% w/w or 2% w/w and at least one of the following optional ingredients such as, solvent, fragrance, preservative, dye, bactericide and filler.

10

Other ingredients which may form part of this invention are disintegrants to provide quick dissolution in water without the need for any stirring or agitation, flowing agents to improve the flowability of the solids during

15 manufacturing, anti-cake and anti-stick agents to reduce the stickiness of the solids to the mould during powder tabletting, binding agents to provide products with good cohesion and adsorbing products which entrap any fragrance that is present.

20

Preferably from 1 to 25g of product is used per machine, ideally from 2 to 22g, and preferably from 10 to 20g.

#### Surfactant

25

Preferred levels of surfactant are from 1 to 40% w/w, ideally 10 to 20% wt and preferably 15 to 19% w/w. Non ionic and anionic surfactants can be used.

30 The non-ionic surfactant is preferably a surfactant having a formula  $RO(CH_2CH_2O)_nH$  wherein R is a mixture of linear, even carbon-number hydrocarbon chains ranging from  $C_{12}H_{25}$  to  $C_{16}H_{33}$  and n represents the number of repeating units and is a number of from about 1 to about  
35 12. Examples of other non-ionic surfactants include higher aliphatic primary alcohols containing about twelve

5 to about 16 carbon atoms which are condensed with about three to thirteen moles of ethylene oxide.

Other examples of non-ionic surfactants include primary alcohol ethoxylates (available under the Neodol tradename 10 from Shell Co.), such as C11 alkanol condensed with 9 moles of ethylene oxide (Neodol 1-9), C12-13 alkanol condensed with 6.5 moles ethylene oxide (Neodol 23-6.5), C12-13 alkanol with 9 moles of ethylene oxide (Neodol 23-9), C12-15 alkanol condensed with 7 or 3 moles ethylene 15 oxide (Neodol 25-7 or Neodol 25-3), C14-15 alkanol condensed with 13 moles ethylene oxide (Neodol 45-13), C9-11 linear ethoxylated alcohol, averaging 2.5 moles of ethylene oxide per mole of alcohol (Neodol 91-2.5), and the like.

20 Other examples of non-ionic surfactants suitable for use in the present invention include ethylene oxide condensate products of secondary aliphatic alcohols containing 11 to 18 carbon atoms in a straight or 25 branched chain configuration condensed with 5 to 30 moles of ethylene oxide. Examples of commercially available non-ionic detergents of the foregoing type are C11-15 secondary alkanol condensed with either 9 moles of ethylene oxide (Tergitol 15-S-9) or 12 moles of ethylene 30 oxide (Tergitol 15-S-12) marketed by Union Carbide, a subsidiary of Dow Chemical.

Octylphenoxy polyethoxyethanol type non-ionic surfactants, for example, Triton X-100, as well as amine 35 oxides can also be used as a non-ionic surfactant in the present invention.

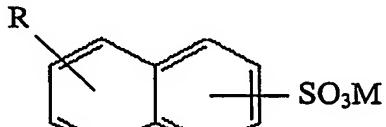
5

Other examples of linear primary alcohol ethoxylates are available under the Tomadol tradename such as, for example, Tomadol 1-7, a C11 linear primary alcohol ethoxylate with 7 moles EO; Tomadol 25-7, a C12-C15 linear primary alcohol ethoxylate with 7 moles EO; Tomadol 45-7, a C14-C15 linear primary alcohol ethoxylate with 7 moles EO; and Tomadol 91-6, a C9-C11 linear alcohol ethoxylate with 6 moles EO.

15 A preferred surfactant is an anionic surfactant. Such anionic surface-active agents are frequently provided in a salt form, such as alkali metal salts, ammonium salts, amine salts, aminoalcohol salts or magnesium salts. Contemplated as useful are one or more 20 sulfate or sulfonate compounds including: alkyl sulfates, alkyl ether sulfates, alkylamidoether sulfates, alkylaryl polyether sulfates, monoglyceride sulfates, alkylsulfonates, alkylamide sulfonates, alkylarylsulfonates, olefinsulfonates, paraffin sulfonates, alkyl sulfosuccinates, alkyl ether sulfosuccinates, alkylamide sulfosuccinates, alkyl sulfosuccinamate, alkyl sulfoacetates, alkyl phosphates, alkyl ether phosphates, acyl sarcosinates, acyl isethionates, and N-acyl taurates. Generally, the alkyl 30 or acyl radical in these various compounds comprise a carbon chain containing 12 to 20 carbon atoms.

Particularly preferred are alkyl naphthalene sulfonate anionic surfactants of the formula:

35



5

wherein R is a straight chain or branched alkyl chain having from about 1 to about 25 carbon atoms, saturated or unsaturated, and the longest linear portion of the alkyl chain is 15 carbon atoms or less on the average, M is a cation which makes the compound water soluble especially an alkali metal such as sodium or magnesium, ammonium or substituted ammonium cation.

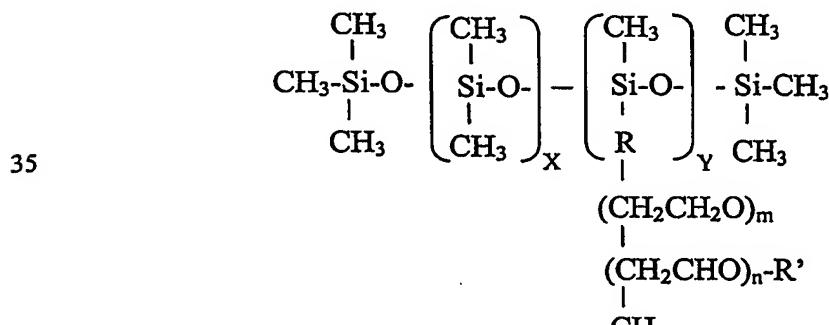
15      Most desirably, the anionic surfactant according to constituent is selected to be of a type that dries to a friable powder. This facilitates their removal from carpets and carpet fibres, such as by brushing or vacuuming.

## Super Wetting Agent

20

A super wetting agent is capable of reducing the surface tension in water to values below 25 mN/m, in the range between 18 and 25 mN/m at concentrations of 0.0001-1%w/v, preferably between 0.001 and 0.1%w/v (when a 15 gram composition is dissolved in 4 litres of water).

Preferred levels in the solid composition are between 0.01 and 10%w/w. Examples of super wetting agents of this invention are silicone glycol copolymers and flurosurfactants. The silicone glycol copolymers are described by the following formula

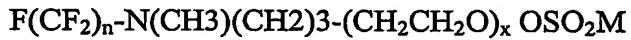
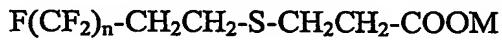


5

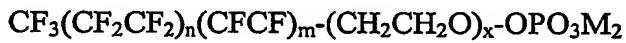
wherein x, y, m and n are each an integer ranging from 0 to 25 (X is preferred between 0-10 and y, m and n between 0-5); R and R' are either a straight or branched alkyl chain having from 1 to 25 carbon atoms, saturated or 10 unsaturated, and the longest linear portion of the alkyl chain is 15 carbon atoms or less on the average.

The fluorinated surfactant is described in the following formulae:

15



20



25

Wherein n, m and x are each integers having a value from 0 to 15; preferred values are between 1 and 12. M is a cation which makes the compound water soluble especially an alkali metal such as sodium or magnesia, armonium or substituted ammonium cation.

#### Antifoaming

Antifoaming agents are an important addition to carpet 30 cleaning compositions of this invention, they are used at a level between 0.01 and 5%w/w. A very high foam level may not allow the carpet cleaning machine to function properly and tends to reduce the mechanical action of the carpet cleaner machine brushes, thus having a detrimental 35 impact on soil removal. Antifoaming agents are also considered important components of this invention.

5 Examples are polydimethylsiloxanes, preferably in combination with hydrophobic silica.

Builders

10 The carpet cleaning composition comprises at least one builder active or better a combination of builders from 1 to 90 % w/w, preferably from 65 to 85 % w/w.

15 Suitable polymer water-soluble compounds include the water soluble monomeric polycarboxylates, or their acid forms, homo or copolymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxylic radicals separated from each other by not more than two carbon atoms, carbonates, bicarbonates, borates, phosphates, and mixtures of any of thereof.

20 The carboxylate or polycarboxylate builder can be monomeric or oligomeric in type although monomeric polycarboxylates are generally preferred for reasons of cost and performance.

25 Suitable carboxylates containing one carboxy group include the water soluble salts of lactic acid, glycolic acid and ether derivatives thereof. Polycarboxylates containing two carboxy groups include the water-soluble salts of succinic acid, malonic acid, (ethylenedioxy) diacetic acid, maleic acid, diglycolic acid, tartaric acid, tartronic acid and fumaric acid, as well as the ether carboxylates and the sulfinyl carboxylates.

30 Polycarboxylates containing three carboxy groups include, in particular, water-soluble citrates, aconitrates and citraconates as well as succinate derivates such as the

10 -

5 carboxymethloxsuccinates described in GB-A-1,379,241, lactoxysuccinates described in GB-A-1,389,732, and aminosuccinates described in NL-A-7205873, and the oxypolycarboxylate materials such as 2-oxa-1,1,3-propane tricarboxylates described in GB-A-1,387,447.

10

Polycarboxylate containing four carboxy groups include oxydisuccinates disclosed in GB-A-1,261,829, 1,1,2,2-ethane tetracarboxylates, 1,1,3,3-propane tetracarboxylates and 1,1,2,3-propane tetracarboxylates.

15 Polycarboxylates containing sulfo substituents include the sulfosuccinate derivatives disclosed in GB-A-1,398,421, GB-A-1,398,422 and US-A-3,936,448, and the sulfonated pyrolysed citrates described in GB-A-1,439,000.

20 Alicyclic and heterocyclic polycarboxylates include cyclopentane-cis,cis,cis-tetracarboxylates, cyclopentadienide pentacarboxylates, 2,3,4,5,6-hexane - hexacarboxylates and carboxymethyl derivatives of polyhydric alcohols such as sorbitol, mannitol and 25 xylitol. Aromatic polycarboxylates include mellitic acid, pyromellitic acid and the phthalic acid derivatives disclosed in GB-A-1,425,343.

30 Of the above, the preferred polycarboxylates are hydroxycarboxylates containing up to three carboxy groups per molecule, more particularly citrates.

More preferred polymers are homo-polymers, copolymers and multiple polymers of acrylic, flourinated acrylic, 35 sulfonated styrene, maleic anhydride, metacrylic, iso-butylene, styrene and ester monomers.

5 Examples of these polymers are Acusol supplied from Rohm & Haas, Syntran supplied from Interpolymer and Versa and Alcosperse series supplied from Alco Chemical, a National Starch & Chemical Company.

10 - The parent acids of the monomeric or oligomeric polycarboxylate chelating agents or mixtures therefore with their salts, e.g. citric acid or citrate/citric acid mixtures are also contemplated as useful builder components.

15 - Borate builders, as well as builders containing borate-forming materials than can produce borate under detergent storage or wash conditions can also be.

- Iminosuccinic acid metal salts.
- Polyaspartic acid metal salts.

20 - Examples of bicarbonate and carbonate builders are the alkaline earth and the alkali metal carbonates, including sodium carbonate and sesqui-carbonate and mixtures thereof. Other examples of carbonate type builders are the metal carboxy glycine and metal glycine carbonate.

25 - Ethylene diamino tetra acetic acid and salt forms.

- Water-soluble phosphonate and phosphate builders are useful for this invention. Examples of phosphate builders are the alkali metal tripolyphosphates, sodium potassium and ammonium pyrophosphate, sodium and potassium and

30 ammonium pyrophosphate, sodium and potassium orthophosphate sodium polymeta/phosphate in which the degree of polymerisation ranges from 6 to 21, and salts of phytic acid.

35 Specific examples of water-soluble phosphate builders are the alkali metal tripolyphosphates, sodium potassium and

5 ammonium pyrophosphate, sodium and potassium and ammonium pyrophosphate, sodium and potassium orthophosphate, sodium polymeta/phosphate in which the degree of polymerization ranges from 6 to 21, and salts of phytic acid.

10

Additional ingredients important for the tabletting of the composition, are selected from the commonly found in detergent tablet compositions, such as described in WO 0233038, and include.

15

Disintegrants

-Suitable cross-linked polymeric disintegrants for use herein include cross-linked starches, cross-linked cellulose ethers, cross-linked polyvinylpyrrolidones, 20 cross-linked carboxy-substituted ethylenically-unsaturated monomers, cross-linked polystyrene sulphonates and mixtures thereof. High preferred are the cross-linked polyvinylpyrrolidones. Suitable cross-linking agents include bi- and multi-functional linking 25 moieties selected from divinyl and diallyl cross-linkers, polyols, polyvinylalcholols, polyalkylenepolymines, ethyleneimine containing polymers, vinylamine containing polymers and mixtures thereof. Alternatively, the vinylpyrrolidone can be cross-linked in-situ by so-called 30 proliferous polymerisation.

Suitable additional disintegrants include:

- a) non-cross and cross linked polymeric disintegrants;
- b) water-soluble hydrated salts having a solubility in 35 distilled water of at least about 25g/100g at 25°C;
- c) effervescent agents; and

5 d) mixtures thereof.

Levels of disintegrant are from 0.1%w/w to 20%w/w, preferably up to 10%w/w.

10 Preferred water-soluble hydrated (fully or partially) salts are selected from hydrates of sodium acetate, sodium metaborate, sodium orthophosphate, sodium dihydrogenphosphate, disodium hydrogen phosphate, sodium potassium tartrate potassium aluminium sulphate, calcium 15 bromide, calcium nitrate, sodium citrate, potassium citrate and mixtures thereof. Particularly suitable materials include sodium acetate trihydrate, sodium metaborate tetrahydrate or octahydrate, sodium orthophosphate dodecahydrate, sodium dihydrogen phosphate 20 dihydrate, the di-, hepta- or dodeca- hydrate of disodium hydrogen phosphate, sodium potassium tartrate tetrahydrate, potassium aluminium sulphate dodecahydrate, calcium bromide hexahydrate, tripotassium citrate monohydrate, calcium nitrate tetrahydrate and sodium 25 citrate dihydrate. In preferred embodiments, the water-soluble hydrated salt is selected from water-soluble mono-, di- tri- and tetrahydrate salts and mixtures thereof. Highly preferred herein is sodium acetate trihydrate, tripotassium citrate monohydrate, mixed 30 alkali-metal citrates containing at least one potassium ion and mixtures thereof. Highly preferred are sodium acetate trihydrate and tripotassium citrate monohydrate.

Where an effervescence system is present it comprises as 35 its components, an acid and a carbonate source, capable

5 of formation of carbon dioxide upon contact with water.

The acid source component may be any organic, mineral or inorganic acid, or a derivative thereof, or a mixture thereof. Preferably the acid source component comprises  
10 an acid or partially salified polymer.

The acid compound is preferably substantially anhydrous or low-hygroscopic and the acid is preferably water-soluble. It may be preferred that the acid source is  
15 overdried.

Suitable acids source components include acidic polymers already described previously as builders, citric, maleic, malic, fumaric, aspartic, glutaric, tartaric succinic or  
20 adipic acid, monosodium phosphate, boric acid, or derivative thereof acidic polymers are especially preferred.

As discussed above, the effervescence system preferably  
25 comprises an alkali source, however, for the purpose of the invention, it should be understood that the alkali source may be part of the effervescence particle or can be part of the cleaning composition comprising the particle, or can be present in the washing liquor,  
30 whereto the particle or the cleaning composition is added.

Any alkali source which has the capacity to react with the acid source to produce a gas may be present in the  
35 particle, which may be any gas known in the art, including nitrogen oxygen and carbon dioxide gas.

5 Preferred can be perhydrate bleaches, including perborate, and silicate material. The alkali source is preferably substantially anhydrous or non-hydroscopic. It may be preferred that the alkali source is overdried. Preferably this gas is carbon dioxide, and therefore the  
10 alkali source is a preferably a source of carbonate, which can be any source of carbonate known in the art. In a preferred embodiment, the carbonate source is a carbonate and bicarbonate salts. Examples of preferred carbonates are the alkaline earth and alkali metal  
15 bicarbonates and carbonates, including sodium or potassium carbonate, bicarbonate and sesqui-carbonate and any mixtures thereof with ultra-fine calcium carbonate such as are disclosed in German Patent Application No. 2,321,001 published on Nov. 15, 1973. Alkali metal  
20 percarbonate salts are also suitable sources of carbonate species, which may be present combined with one or more other carbonate sources.

25 The carbonate and bicarbonate preferably have an amorphous structure. The carbonate and/or bicarbonates may be coated with coating materials. The molecular ratio of the acid source to the alkali source present in the particle core is preferably from 50:1 to 1:50, more preferably from 20:1 to 1:20 more  
30 preferably from 10:1 to 1:10, more preferably from 5:1 to 1:3, more preferably from 3:1 to 1:2, more preferably from 2:1 to 1:2.

Flow agents

35 Examples are silica powder, talc and metal stearates. They improve the flowability of the powder during

5 manufacturing and they reduce the stickiness of the powder to the mould during powder tabletting

#### Binding agents

Examples of binders are polyethylene and polypropylene  
10 glycol with an average molecular weight ranging from 100 to 10000 and non cross-linked starches, cellulose ethers, polyvinylpyrrolidones, carboxy-substituted ethylenically-unsaturated monomers, polystyrene sulphonates and mixtures thereof. High preferred are the  
15 polyvinylpyrrolidones.

#### Solvents

The solvent constituent of the inventive compositions include one or more alcohols, glycols, acetates, ether  
20 acetates and glycol ethers. Exemplary alcohols useful in the compositions of the invention include C2-C8 primary and secondary alcohols which may be straight chained or branched. Exemplary alcohols include pentanol and hexanol. Exemplary glycol ethers include those glycol  
25 ethers having the general structure Ra-O-Rb-OH, wherein Ra is an alkoxy of 1 to 20 carbon atoms, or aryloxy of at least 6 carbon atoms, and Rb is an ether condensate of propylene glycol and/or ethylene glycol having from 1 to 10 glycol monomer units. Preferred are glycol ethers  
30 having 1 to 5 glycol monomer units. By way of further non-limiting example specific organic constituents include propylene glycol methyl ether, dipropylene glycol methyl ether, tripropylene glycol methyl ether, propylene glycol n-propyl ether, ethylene glycol n-butyl ether,  
35 diethylene glycol n-butyl ether, diethylene glycol methyl ether, propylene glycol, ethylene glycol, isopropanol,

17 -

5 ethanol, methanol, diethylene glycol monoethyl ether acetate and particularly useful is , propylene glycol phenyl ether.

Process manufacturing:

10

The powder raw materials can be mixed directly or liquid spraying process can be required for certain raw materials such as fragrances, super wetting agents and antifoaming agents.

15

A wet granulation process could be required so that the fine powder ingredients are granulated with a water based formulation containing several ingredients as for example antifoaming agent, superwetting agent, dyes and 20 fragrance. After the granulation the wet powder is dried, then sieved and mixed with other coarse powders.

25 The tablets may be manufactured by using any compacting process, such as tabletting, briquetting, or extrusion, preferably tabletting. Suitable equipment includes a standard single stroke or a rotary press (such as Courtoy.RTM., Korsch.RTM., Manesty.RTM., or Bonals.RTM.).

30 Examples of suitable tablet shapes are cylindrical, spherical and cubic.

The compaction pressure used for preparing these tablets is in the range 1000-20000 kN/m<sup>2</sup>, preferably between 1500 and 10000 kN/m<sup>2</sup>.

35

5 Evaluation Test

The tablets have been evaluated in terms of surface tension when dissolved in deionised water, dissolution time in warm water ( $T=40^{\circ}\text{C}$ ), remaining residue after 10 dissolution and soil removal performance versus Resolve<sup>TM</sup> for steam machine taken as a reference of the machine carpet cleaner products.

SURFACE TENSION MEASUREMENTS:

15

The surface tension has been measured with a surface tensiometer, by the ring method. A platinum du Nuoy ring is immersed in 12 French degree water solution maintained at  $20^{\circ}\text{C}$ , where previously has been dissolved a tablet.

20 The ring is taken out slowly from the liquid. When the ring is near the air/liquid interface, it is balance by the tensiometer. The maximum force versus area gained before breaking the liquid film formed is the surface tension of the liquid.

25

The lower the surface tension expressed in  $\text{mN/m}$ , the better the formula performance in terms of wettability on carpet surface.

30 A surface tension target value for the composition is below 28  $\text{mN/m}$ .

DISSOLUTION AND RESIDUE EVALUATION TEST:

35 One tablet of 15 grams is put in a beaker containing 41 of warm water ( $T=40^{\circ}\text{C}$ ). The effervescent effect, the

5 dissolution time as well as the remaining residue quantity are considered and recorded.

A table with all parameter ratings is reported below:

Dissolution rating	Effervescent effect	Dissolution time	Residue
0	No effect	More than 30 minutes	More than 20% wt
1	No effect	More than 10 minutes	More than 20% wt
2	Low	More than 10 minutes	More than 10% wt
3	Medium	Between 5 and 10 minutes	More than 10% wt
4	Medium	Between 5 and 10 minutes	Between 5 and 10 % wt
5	Strong	Between 5 and 10 minutes	Between 5 and 10 % wt
6	Strong	Less than 5 minutes	Between 5 and 10 % wt
7	Strong	Less than 5 minutes	Less than 5 % wt

10

The higher the rating number, the better the dissolution.

SOIL REMOVAL TEST:

15 This method has been designed for the evaluation of soil removal performance of extraction cleaner formulations.

The soil has the following composition:

20 -

	- Soil components:	% by weight
	- Peat Moss	47.7
	- Cement	21.4
	- Kaolin clay	8.0
10	- Silica	8.0
	- Red Iron oxide	1.3
	- Charcoal	12.6
	- Mineral oil	1.0

15 A white nylon carpet is used for the test.

The carpet is soiled with 5 grams of standard soil. The soil is applied 1 gram once by strainer. The soiled carpet is then put with 4 kg of steel beads in the jar mill and stirred for 30 minutes at 56 rpm.

20 They are dissolved in warm tap water ( $T = 40^{\circ}\text{C}$ ), one 15 grams per 4 l of water. The Resolve for steam machine is diluted according to its labelling instructions.

Carpet is cleaned with appropriate machine/product using 4 wet strokes (dispensing solution) and 2 dry strokes

25 (vacuuming up solution).

Carpet swatches are placed in a dark room temperature chamber ( $25^{\circ}\text{C} / 50\%\text{RH}$ ) for 24 hours while they dry.

30 The cleaning performance is evaluated by measuring the carpet with a portable spectrophotometer before soiling, after soiling and after the cleaning process. The result is reported as soil removal percentage.

EXAMPLES:

5 The fined powder are typically granulated with a water formulation, then dried and mixed with the remaining coarse powder ingredients Examples of compositions forming a part of the present invention are set below in Table 1 with the various components identified in Table  
 10 2.

Table 1

Components	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5	Ex 6
	%	%	%	%	%	%
Baypure CX100	7.28					
Baypure DS100	0.82					
Citric acid anhydrous DC F20	14.00	14.00			7.00	
Bicar-TEC 27/50 G	15.00	15.00	15.00	15.00	13.00	15.00
Tri-sodium citrate dehydrate F6000	9.20	18.30	8.80	8.80		7.82
Sodium carbonate anhydrous	26.00	26.00	26.00	24.80	33.30	25.32
PEG 6000	4.00	4.00	4.00	4.00	4.00	2.50
Petro 11 powder	16.50	16.50	16.50	16.50	16.50	16.85
Syntran DX 102-2-3	1.00					
Acusol WE			23.50	24.70	20.00	24.70
Disintex 75	1.00	1.00	1.00	1.00	1.00	1.00
DC Q2-5211	4.00	4.00	4.00	4.00	4.00	4.09

Dye						0.02
Fragrance						0.50
Sipernat 22 S						1.00
BF 20 PLUS	1.20	1.20	1.20	1.20	1.20	1.20

5

Table 2

Component	Description of component
Baypure CX100	Imminodisuccinic acid sodium salt from Bayer
Baypure DS100	Polyaspartic acid sodium salt from Bayer
Citric acid anhydrous DC F20	Starch coated citric acid from Jungbunzlauer
Bicar-TEC 27/50 G	Sodium bicarbonate from Solvay
Tri-sodium citrate dehydrate F6000	Tri-sodium citrate dehydrate from Jungbunzlauer
Sodium carbonate anhydrous	Sodium carbonate anhydrous light from Solvay
PEG 6000	Polyethylene glycol 6000 from Medivete Padana
Petro 11 powder	Sodium alkyl naphthalene sulfonate from Witco
Syntran DX 102-2-3	Acrylic polymer from Interpolymer
Acusol WE	Acrylic polymer from Rohm&Haas
Sipernat 22 S	Silica powder from Degussa
Fragrance	Proprietary fragrance from various suppliers
Dye	Proprietary dye from various suppliers
Disintex 75	Cross-linked polyvinyl polypyrrolidinone from ISP
DC Q2-5211	Methyl(propylhydroxide, ethoxylated)bis(trimethylsiloxy) silane from Dow Corning

BF 20 PLUS	Silicone emulsion from Dow Corning
5	

EXAMPLE RESULTS:

The tablet example products have been compared with  
 10 Resolve™ for steam machine carpet cleaner in terms of  
 soil removal performance. Oxi Clean™ multi specialist  
 powder from Orange Glo Int. and Bissell™ booster powder  
 from Bissell have been used for comparing the dissolution  
 performance.

15

Results for table 1 formulations:

Product	Surface tension (mN/m)	Dissolution rating	Soil Removal percentage
Ref 1 (Resolve steam)	29	-	24.0
Ref 2 (Oxi clean)	-	0/1	-
Ref 3 (Bissell)	-	1	-
Ex 1	-	7	26.2
Ex 2	27.8	6	24.0
Ex 3	-	6	26.5
Ex 4	24.5	7	26.8
Ex 5	25.6	7	25.1
Ex 6	24.0	7	28.9

## 5 CLAIMS

1. A method of cleaning a soiled carpet the method comprising adding to the reservoir of a carpet cleaning machine a water soluble solid product comprising a carpet cleaning composition, adding water to the reservoir of the carpet cleaning machine prior to or after adding the water soluble solid product, and operating the carpet cleaning machine on the soiled carpet.
- 15 2. A method as claimed in claim 1 wherein the water soluble solid product is added to the reservoir of the carpet cleaning machine prior to adding the water.
- 20 3. A method as claimed in claim 2 or claim 3 wherein the water soluble solid product is a powder, granule, pellet or a tablet.
- 25 4. A method as claimed in claim 3 wherein the solid is a tablet.
5. A method as claimed in any claim from 1 to 4 wherein the carpet cleaning composition comprises at least one surfactant and at least one super wetting agent and wherein the combined effect of the surfactant and the super wetting agent in the composition is capable of reducing the surface tension of water below 28 mN/m when 15 g of the composition is dissolved in 4 litres of water.

5 6. A water-soluble carpet cleaning composition in solid  
form comprising at least one surfactant and at least  
one wetting agent wherein the combined surface  
tension effect of the surfactant and the super  
wetting agent in the composition is capable of  
10 reducing the surface tension of water below 28 mN/m  
when 15 g of the solid product is dissolved in 4  
litres of water

15 7. A water-soluble carpet cleaning composition in solid  
form as claimed in claim 6 which additionally  
comprises a builder, a disintegrant, an antifoaming  
agent, a solvent and a fragrance.

20 8. A water-soluble carpet cleaning composition in solid  
form as claimed in either claim 6 or 7 which is in a  
tablet form.